

MACRO STRESS MAPPING ON THIN FILM BUCKLING

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INTRODUCTION

Thin films deposited by Physical Vapor Deposition techniques on substrates generally exhibit large residual stresses which may be responsible of spontaneous detachment of the film from the substrate (stress relaxation) and in the case of compressive stresses, thin film buckling. Although these effects are undesirable for future applications, one may take benefit of it for thin film mechanical properties investigation [1-4]. Since the 80's, a lot of theoretical works have been done to develop mechanical models and calculations (elasticity of thin plates, fracture mechanic) with the aim to get a better understanding of driven mechanisms giving rise to this phenomenon and thus to propose solutions to avoid such problems. Nevertheless, only a few experimental works have been done on this subject to support these theoretical results and nothing concerning local stress/strain measurement mainly because of the small dimension of the buckling (fig. 1). In this experiment, we propose to use micro beam x-ray diffraction available on synchrotron radiation sources as a local probe (spatial) for stress/strain analysis of thin film buckling. The main objectives are to apply x-ray micro beam diffraction in first for determining macro residual stresses at the top of the buckle (comparison with adherent region on the film) in different systems (W, Mo, Au on silicon) and in a second step for scanning the buckling with the smallest x-ray beam size in order to realize macro stress mapping.

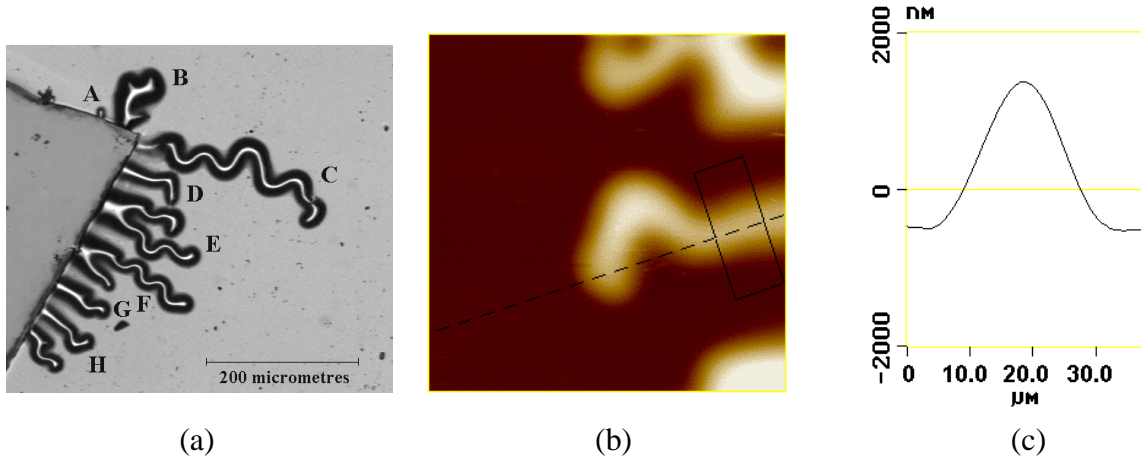


Figure 1 : Wrinkles observed by optical microscopy (a) close to a step realised during the deposition process for film thickness measurement and AFM images (b) of buckling noted D on (a) and corresponding cross section, (c) perpendicular to the propagation direction.

EXPERIMENTAL METHOD

Among the most widely used method to determine the stress level in thin films, x-ray diffraction (XRD) is phase selective and the unique non destructive technique which allows to determine both the mechanical and microstructural state of the diffracting phases. Indeed, the distance between atomic planes is used as an internal strain gauge. For polycrystalline samples, the

measurement of the diffraction peak position shift using $\sin^2\psi$ method allows to extract the stress tensor and the stress free lattice parameter [5]. However, x-ray diffraction is difficult to use in low dimensional systems because the diffracted intensities are weak due to the reduced thicknesses and nanocrystalline character of such materials. These problems may be solved using intense x-ray sources such as synchrotron radiation (S.R.). In addition to the high flux characteristic of S.R. facilities, the wide wavelength spectra and the optics (micro beam) which are available on beam lines (3rd generation SR only) allow to perform specific XRD experiments which are not possible with classical x-ray sources in laboratories.

The 7.3.3.1 Microdiffraction beam line at ALS provides a reduced spot size less than $1\text{ }\mu\text{m}^2$ (Kirkpatrick Baez mirrors) with high flux for white or monochromatic radiation (4-crystals channel cut monochromator: 6-14 KeV). These performances are unique and perfectly adapted to our project [6]. In our samples, the grain size ($\leq 10\text{ nm}$) is smaller than the x-ray beam one and macro-strains are of about 0.5-1 %. The diffraction pattern recorded with a 2D CCD detector is composed of different rings which allow to extract (using specific software - to be developed) the in-plane stress without any tilt of the samples (the ψ description is contained in the 2D pattern and the angular resolution is enough to appreciate such large macro-strains). Accurate spatial spot localization on the sample surface is achieved from markers delimiting the region of interest. A precise goniometer allows XRD measurements in reflection mode and an X-Y translation stage is used for scanning the sample surface.

FEASIBILITY OF THE EXPERIMENT

Preliminary microdiffraction experiments have been successfully done in June 2001. 630 nm thick gold films deposited on silicon (100) substrates covered with native oxide have been chosen for these measurements. The delamination of the thin film is evidenced on figure 2 (a) and an individual buckle is shown on figure 2 (b); its shape corresponds to a portion of a sphere. The position of the buckle (x,y) is determined from the step corner coordinates (x0,y0) which have been measured by X-ray fluorescence (white beam). This step has been realized during the film deposition.

The calibration of the 2D diffraction diagram is done from the diffraction of the single crystal silicon substrate using white x-ray beam. The diffraction pattern obtained in an adherent (flat) region of the gold film is shown on figure 3. Because of the $\langle 111 \rangle$ texture of the film, we observe maximum intensities on each two rings for particular pole directions X. During the scan of the buckle A with a $10\text{ }\mu\text{m}$ step, the position of the two maximum intensities (X+ and X-) moves along the ring because of the rotation of the normal to the film surface around x and y axis. Thus, from the diffraction pattern, one may deduce the position of the beam on the buckle and the corresponding strains in this region. A comparison between the diffraction diagrams obtained in flat regions and at the top of the buckle indicates a stress relaxation.

A complete interpretation of the data is still under progress because specific tools and methodologies have to be applied for diffraction pattern analysis. Accuracy and sensibility are the two main points to be considered for strain variation during the scan. Furthermore, Finite Element calculations have been engaged for such spherical gold buckles. Further experiments will concern not only metallic thin films but also structural biomaterials such as diamond coatings on Titanium based alloys (TA6V) which are promising candidates for medical

prostheses. Our laboratory is engaged in a French government program called A.C.I. with two other French laboratories. It concerns in particular, the influence of residual stresses (about -5 GPa in C-diam) on the mechanical behavior of such systems.

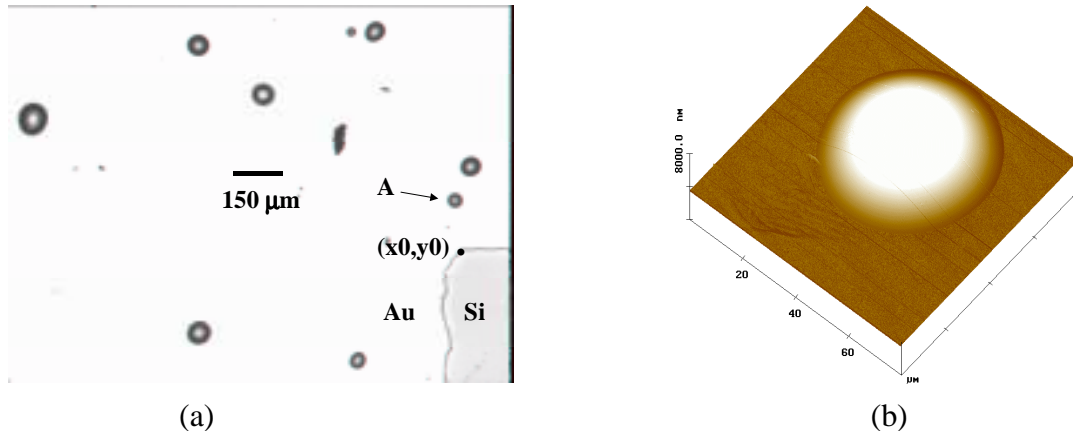


Figure 2. 630 nm gold film sputter deposited on Si substrate: (a) optical image of the sample surface and (b) AFM image of the buckle noted A on fig. (a); the in plane width is around $40\ \mu\text{m}$ and the height of $1.2\ \mu\text{m}$.

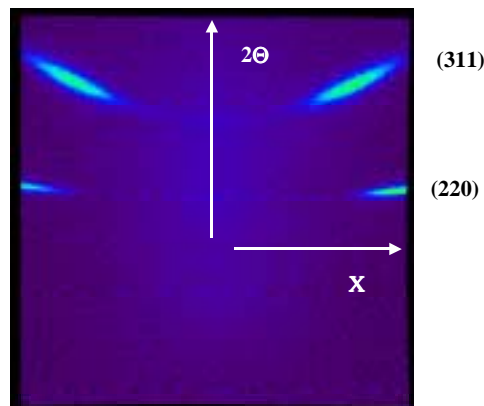


Figure 3: 2D X-ray diffraction diagram obtained on an adherent gold film region with an X-ray energy of 5.7 keV, a spot size on the sample of $3 \times 3\ \mu\text{m}^2$ and a recording time of 300 sec.

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